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FINAL REPORT, BARRIER MATERIAL (HEAVY DUTY)

MAR 12, 1957

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CONFIDENTIAL

March 12, 1957

F I N A L R E P O R T

ON

BARRIER MATERIAL (HEAVY DUTY)

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Introduction

The objective of the work performed under this task was to test and evaluate a special heat sealable, heavy duty barrier material for possible use as a packaging medium. As a control, packages of heat sealable aluminum foil were prepared and then subjected to the hot dip plastic process to obtain a protective coating.

Past experience has shown that after sealing the heavy duty material, delamination occurs between the two layers of aluminum foil. Tests were performed to obtain a suitable material which could be applied to the exposed edges and prevent this delamination. Under the terms of our proposal, dated March 29, 1956, a foot operated heat sealer with Teflon adapter, manufactured by the Mercury Heat Sealing Equipment Company of Philadelphia, Pennsylvania, was purchased at the request of the Project Officer. This sealer is a jaw type device with the jaws heated by a cartridge and thermostatically controlled. Pressure control and dwell time are manually
spring regulated
controlled. An evaluation of the heat seal on the heavy duty barrier material was performed to determine the best method of closure.

Evaluation of Heat Seal on Heavy Duty Material

Small samples of the heavy duty barrier material were used to obtain seals starting at a temperature of 300 Deg. F and proceeding to the point at which a satisfactory seal was obtained. The temperature of the jaws was raised in increments of 25 Deg. F to 425 Deg. F. At this temperature, and with a dwell time of 9 seconds, suitable seals were obtained. The rather high temperature and long dwell time was required due to the thickness of the material and the conductivity of the two layers of foil.

The sealable foil required a temperature of 275 Deg. F and a dwell time of 3 seconds.

Criteria for a Suitable Coating

The following criteria were considered in order to determine which material would be the most suitable of the coatings investigated.

1. The coating should not break down under the surveillance conditions listed below. This is the most important criterion to be observed.
2. There must be a strong bond between the coating and the outer layer of the barrier material.
3. The material must be easy to apply by either a brushing, dipping, or spraying method.

4. The preliminary preparation of the coating should not require any elaborate mixing, with direct application of the material, as received from the manufacturer, preferred.

5. The substance must have a short curing time at room temperature or be of such a nature that the curing time can be accelerated by being exposed to moderately high temperature (150 Deg. F - 175 Deg. F).

   Too hot for some items, e.g., A.C. Delay

6. A pot life of roughly 4 hours would be satisfactory since the preparation could be made prior to a normal 4 hour production run.

<u>Experimental Procedure</u>

There are many plastics which fill the general requirements of the above criteria, however, three were selected which were representative of plastics currently on the market. They were, a phenolic type plastic (Catalin Resin 9585) used for tool making, a modified urea plastic, (Catalin Resin 8666) which has good resistance to cracking, and a resorcinol plastic (Catalin Resin 736) which produces waterproof bonds. All three are manufactured by Catalin Corporation of America.

Volatile solvent type compounds which were investigated were Glyptal, an alkyd-resin product manufactured by General Electric Company, Schenectady, New York and a common household shellac.

Packages of the heat sealable, heavy duty, barrier material were prepared and a piece of mild steel was added to each package so that the vapor transmission, if any, would be indicated. The packages were sealed using the Mercury Heat Sealer at a temperature of 415 Deg. F and a dwell time of approximately 9 seconds.

Each of the coatings was used on a series of packages which were placed in the following surveillance conditions:

1. Accelerated Aging

   Items were subjected to 160 Deg. F and 90 per cent relative humidity for a period of two weeks.

2. Cycling

   Items were subjected to four consecutive cycles at the following conditions:

   4 hours @ 120 Deg. F to 130 Deg. F and 90% R.H.
   2 hours @ 70 Deg. F to 90 Deg. F and 90% R.H.
   2 hours @ 0 Deg. F to -10 Deg. F or lower
   16 hours @ 35 Deg. F to 50 Deg. F and 20% R.H.

3. Safe Storage Test

   Items were subjected to 160 Deg. F and 22% R.H. for 24 hours then examined for protection. New items were subjected to -60 Deg. F for 24 hours, then tested for protection. All testing was done after items reached ambient temperature.

4. <u>Temperature Limits Test</u>

   Items were subjected to 125 Deg. F and 22% R.H. for 24 hours and then examined for protection at test temperature. New items were subjected to -40 Deg. F for 24 hours, then inspected for protection at test temperature.

5. <u>Salt Fog Test</u>

   This test was conducted in accordance with MIL Specification 57-0-2C for one week. A daily cycle of 16 hours salt fog spray and 8 hours open to atmosphere was maintained.

6. <u>Water Submergence</u>

   Packages were submerged in 10 feet of water for 2 weeks.

Experimental Results

A. Phenolic Plastic

1. The protection from delamination after all surveillance testing was good, however, on rough handling the coating cracked and could be pealed back quite easily.

2. Adhesion to the nylon backing of heat sealable, heavy duty barrier material was poor. The coating was quite easily pealed back.

3. Preparation involved some difficulty. In preparing the plastic, definite proportions of base and

accelerator had to be adhered to in order to avoid altering the pot life and curing time.

4. Application was somewhat difficult. In order to achieve the proper thickness without altering the pot life several applications were necessary. The plastic had a tendency to run down the sides of the package, which could cause great difficulty in production line packaging with units sticking to one another and to drying racks.

5. Curing time was one hour. The only means of accelerating this time is chemically, and this would shorten the pot life.

6. Pot life is 2 hours.

B. Urea Plastic

1. The plastic offered almost no protection at all from delamination after any of the surveillance tests. Delamination occurred at the aluminum foil lamination to a large extent after being subjected to the cycling, accelerated aging salt fog tests to a high degree. Delamination occurred to a lesser degree in temperature limits and safe storage tests. On the results obtained from this series of tests this material could not be used under any circumstances.

2. The adhesion was poor along the edges of the package.

3. Preparation involved problems similar to those encountered with the phenolic plastic.

4. Application was difficult because the plastic was so thin. It was almost impossible to procure a thick coating, even with numerous applications. The plastic was applied with a small brush.
5. Curing time was 1 hour.
6. Pot life was 2 hours.

C. Resorcinol Plastic

1. Delamination occurred at the corners and in some other parts of the edge, however, the degree of delamination was not as serious as that of the urea plastic after accelerated aging, cycling and salt fog tests. Results from other surveillance tests were satisfactory.
2. The resorcinol gave poor adhesion to the edges, however, it did adhere well to the nylon backing.
3. Preparation was similar to (A) and (B) above.
4. Application - the substance was not too viscous and only a very thin coat was obtained even after several applications.
5. Curing time was 45 minutes.
6. Pot life was over 3 hours.

D. Glyptal

1. The protection from delamination was only fair.
2. Adhesion to the nylon backing was good, but the adhesion to the edges of the package was poor.

3. No special preparation is required and this sample can be applied directly as received from the supplier.
4. Glyptal was applied with a small brush and it gave a reasonably thick coat.
5. The curing time was 30 minutes at room temperature and was accelerated to 15 minutes when placed before an electric hot air blower.
6. Glyptal has an indefinite pot life. Acetone was added as needed to maintain the desired viscosity.

E. Shellac

1. The protection from delamination was excellent after all of the surveillance tests. When handled rough after setting the sealer cracked very severely.
2. Adhesion to the nylon backing was good but the adhesion to the edges of the package was poor.
3. No special preparations were required. The shellac can be used directly from the can.
4. Shellac was applied with a small brush.
5. The curing time was 10 minutes at room temperature and could be accelerated by heat.
6. Shellac has an indefinite pot life and requires only the addition of solvent to maintain the proper working consistency.

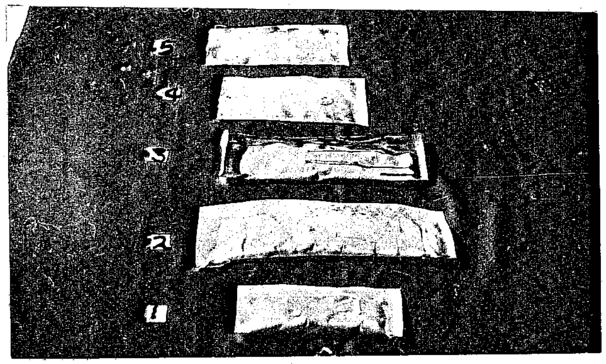

Figure 1

Figure 1 is representative of the packages made up with the plastics and the substances employing a volatile solvent. They are:

1) Phenolic plastic
2) Urea plastic
3) Resorcinol plastic
4) Glyptal
5) Shellac

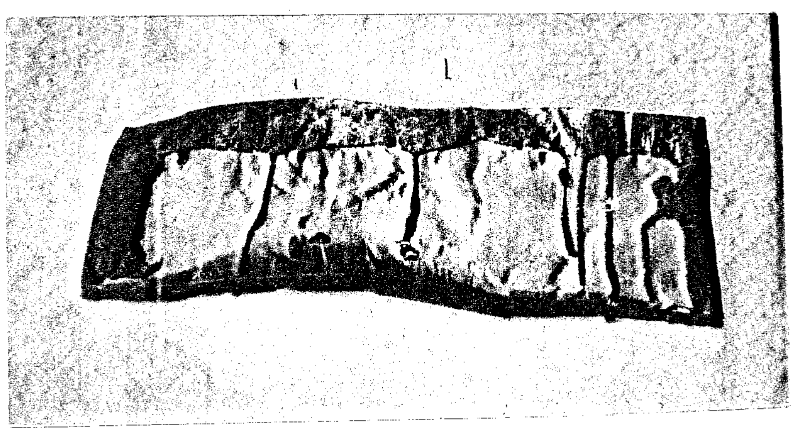

Figure 2

Figure 2 is a view of a package coated with the urea plastic. It demonstrates the problems encountered

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with the plastic flowing down the sides of the packages which would cause adhesion to foreign materials, such as drying jigs, during the curing time.

The upper right hand corner of the package shows the extent to which this sample cracked.

Remarks

The phenolic plastic and the urea plastic showed many cracks when taken from the surveillance tests. The resorcinol, though it did not crack, did not give sufficient adhesion to the exposed edges. Glyptal and shellac also cracked when taken from tests and gave poor adhesion to the exposed edges. When the packages were flexed and tested for rough handling the coating on the edges would crack and break. After this test, delamination, to a serious degree, became quite evident.

From the above results it was obvious that some other sealer which would offer a more pliable coating and which would have better adhesive qualities would be required. This led to the testing of a group of rubber sealers.

Volatile Solvent Rubber Base Cements

As with the plastics, there are many rubber materials which fill the general requirements, however, five rubbers which were representative of a large group of commercially available products were selected. They were Presstite Sealer, a butadiene rubber manufactured by the Presstite Engineering Company, St. Louis, Missouri; XL8 Rubber Cement, manufactured by R. H. Products Company, Acton, Massachusetts; Neoprene Bonding Cement, a product of Greene Rubber Company, Cambridge, Massachusetts; 3M Adhesive, manufactured by Minnesota Mining and Manufacturing Company, Detroit, Michigan and Black Tuffy, a thiokol rubber (polysulfide plastic) manufactured by Permaflex Mold Corporation, Columbus, Ohio.

Black tuffy in the strict sense does not fall into this category since it employs a catalyst while the other four products employ a volatile solvent.

Test packages were prepared in the same manner as described above and were subjected to the same surveillance tests.

<u>Results</u>

A. Presstite Sealer

1. The protection from delamination was excellent after being exposed to all of the surveillance tests.
2. Adhesion was excellent.
3. No special preparation was required except for thinning occasionally with methyl ethyl ketone.
4. A rather thick coat was applied using a tongue depressor as an applicator. There was no difficulty with this sample flowing down the sides.
5. The curing time was 45 minutes. This time could be accelerated by placing the package before a hot air blower.

6. Pot life is indefinite.

B. XL8 Rubber Cement

1. The protection from delamination was excellent. This material was not affected by the surveillance tests.
2. Adhesion was excellent.
3. No special preparation was required.
4. A very thick coating was obtained in one application using a small brush.
5. Curing time was 30 minutes at room temperature.
6. The pot life is indefinite.

C. Neoprene Bonding Cement

1. Protection from delamination was excellent after exposure to all surveillance testing.
2. Adhesion was excellent.
3. No special preparation was required.
4. A fairly thick coat was obtained using a small brush.
5. Curing time was thirty-five minutes at room temperature.
6. Pot life of neoprene is indefinite. Practically any solvent can be used to obtain proper consistency.

D. 3M Adhesive

1. Protection from delamination after all surveillance tests was excellent.

2. Adhesion was good.

3. No special preparation is required.

4. A good coat was obtained using a tongue depressor as an applicator.

5. Curing time was ten minutes at 100 Deg. F.

6. 3M adhesive has an indefinite pot life.

E. Black Tuffy

1. The protection from delamination after all surveillance tests was excellent.

2. Adhesion was good, a few air bubbles were found on some of the packages, but these could be eliminated at the time of application.

3. Preliminary preparation was required with close tolerances of additives necessary to obtain suitable properties.

4. The material was easily applied using a small brush.

5. Curing time was 30 minutes at 100 Deg. F.

6. Pot life was over 4 hours.

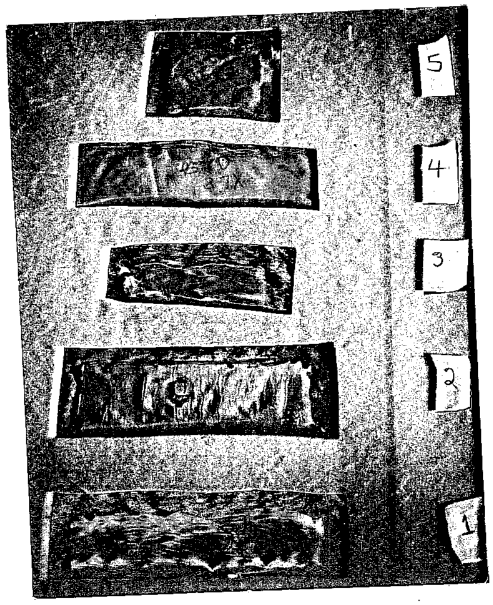

Figure 3

Figure 3 demonstrates a series of packages prepared with the rubber substances. They are:

1) Presstite Sealer
2) Black Tuffy
3) XL8 Rubber Cement
4) 3M Adhesive
5) Neoprene Bonding Cement

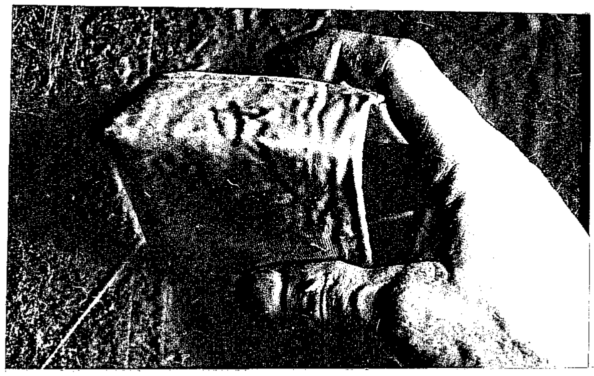

Figure 4

Figure 4 demonstrates the flexibility of the rubber sealers. This is a view of the XL8 rubber cement which was bent back and forth several times. This package returned to its original configuration without damage to the seal. This type of handling could be given to all of the rubber samples without causing any cracking to the sealer.

Remarks

The Presstite Sealer, XL8 rubber cement and Neoprene Bonding Cement all gave excellent results in all respects. The 3M adhesive when handling roughly at extremely low temperature (below -25 Deg. F) cracked, however, if the package was allowed to reach room temperature before the rough handling test was performed, complete flexibility was again exhibited.

Recommendations

The rubber based sealers investigated under this contract, or their equivalent should be used to overcome the problem of delamination. This type of sealer offers excellent protection and in addition to their practicability are quite inexpensive and are readily obtained.

The general run of plastics should be avoided for this application because when they are cured they become quite brittle and crack very easily. However, one group of plastics could be considered as a secondary material to the satisfactory samples mentioned above. This group is the rubber base plastics, which includes the Black Tuffy sample.

This group has good to excellent properties for coating and resistance to acids, alkalies, water, etc. However, the objection to these materials are the preliminary preparation and the short pot life.

Any reputable rubber base cement, which can be applied directly from the can would appear satisfactory to prevent delamination.

Table I lists sealers which were examined in this investigation with the experimental results summed in tabular form.

Evaluation of the sealers was made on a basis of excellent, good, fair, and poor.

TABLE I

Sealer	Protection Surveillance	Adhesion	Preparation and Application	Curing Time (mins.)	Pot Life (hrs.)
Phenolic plastic	Good	Poor	Poor	60	2
Urea Plastic	Poor	Poor	Poor	60	2
Resorcinol plastic	Fair	Poor	Poor	45	3
Glyptal	Fair	Fair	Good	30	I**
Shellac	Good	Good	Excellent	10	I**
Presstite	Excellent	Excellent	Excellent	45*	I**
XL-8	Excellent	Excellent	Excellent	30	I**
Neoprene	Excellent	Excellent	Excellent	35	I**
3M Adhesive (EC968)	Excellent	Excellent	Excellent	10*	I**
Black Tuffy	Excellent	Good	Good	30*	4

*Curing time accelerated by means of a forced hot air electric blower.

**I - Indefinite pot life.

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Conclusions

1. The plastics which were tested exhibited poor properties for this particular application. The coatings which were applied to the packages were too thin to offer sufficient protection along the exposed edges of the package and in order to obtain proper thicknesses repeated applications of the material would be necessary.
2. The plastics do not have the ability to withstand rough handling since the thin coating cracks quite easily.
3. The application of the plastics does not lead to neat appearing units because of the viscosity. To obtain a more suitable flow of the material would render the plastic unsuitable with respect to pot life and curing time.
4. The shellac coating shows poor resistance to rough handling because of its brittleness. However, it is quite easy to control the viscosity with alcohol and is easily applied. The curing time is very good and could be used in extreme cases to obtain a suitable bond.
5. The rubber base materials are all quite satisfactory since they conform to the criteria for a suitable sealer established earlier in this report. They can be easily applied, prepared, offer good protection

and can be worked to any desired consistency merely by adding solvent.

Several packages of the four suitable materials were prepared and placed in the seven long term [redacted]. The 25X1
results of this extended evaluation will be reported at a later date under Contract No. RD-88, Task No. 8 which is presently under investigation.

19 March

Walt + **WE** for info

~~Joe~~ Jt Please return to [redacted] 25X1

I think this is a pretty good report. The information is rather late, now that nylon material is obsolescent, but it is still of value in our use of the remainder of the nylon material.

Howie

Joe - another report for the Reports file. This is still [redacted] 25X1

ED 39 right?

Walt
27 Mar 57

March 13, 1957

Dear Howie:

Transmitted herewith is our final report on "Barrier Material (Heavy Duty)," Task 1210-E-6h. We hope our findings and conclusions will be very useful in your work. If you have any questions concerning this task, please do not hesitate to call on us.

Regards,

Frank

Frank

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Enclosures 4